



## INTERNATIONAL APPLICATION PUBLISHED UNDER THE PATENT COOPERATION TREATY (PCT)

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<b>(54) Title:</b> DURABLE PRESS/WRINKLE-FREE PROCESS  <b>(57) Abstract</b> <p>Cellulosic fiber-containing fabrics are made wrinkle resistant by a durable press wrinkle-free process which comprises treating a cellulosic fiber-containing fabric with formaldehyde, a catalyst capable of catalyzing the cross-linking reaction between the formaldehyde and cellulose and a silicone elastomer, heat-curing the treated cellulose fiber-containing fabric, preferably having a moisture content of more than 20 % by weight, under conditions at which formaldehyde reacts with cellulose in the presence of the catalyst without a substantial loss of formaldehyde before the reaction of the formaldehyde with cellulose to improve the wrinkle resistance of the fabric in the presence of a silicone elastomeric softener to provide higher wrinkle resistance, and better tear strength after washing, with less treatment.</p> <p><i>3, 1, 2, 13, 14</i></p>		

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## DURABLE PRESS/WRINKLE-FREE PROCESS

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CROSS REFERENCE TO RELATED APPLICATION

This application claims benefit under 35 USC 119(e) of prior pending application 60/046,298 filed May 13, 1997.

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BACKGROUND OF THE INVENTIONField of invention

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This invention relates to a durable press/wrinkle-free process for cellulosic fiber-containing fabrics and more particularly to a process which permits high treatment level amounts of formaldehyde and catalysts to impart wrinkle resistance to the cellulosic fiber-containing fabrics while reducing the loss in both tensile and tear strength normally associated with such treatment processes.

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Description of related art

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There are a number of known process for treating cellulosic fiber-containing fabrics, such as cotton-containing fabrics, to make them wrinkle-free. These treatment processes include resin or polymer treatment of the fabric, but these are costly and unsatisfactory. Another process for treating cellulosic fiber-containing products relies on formaldehyde to provide durable crosslinking of the cellulose molecules and to thereby impart durable crease

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resistant and smooth drying characteristics to these products. However, problems have been encountered with the known processes. A simple, reproducible, completely satisfactory low-cost formaldehyde durable press process has not yet been achieved.

5           It has long been known to treat cellulosic materials with formaldehyde, as is evidenced by U.S. Patent Number 2,243,765. This patent describes a process for treating cellulose with an aqueous solution of formaldehyde containing a small proportion of an acid catalyst under such conditions of time and temperature that the reaction is allowed to approach its equilibrium.

10       It is further stated that, in carrying out this process, the proportion of the solution of formaldehyde to the cellulose must be at least such that the cellulose is always in a fully swollen state. It is also stated that the time and temperature of the treatment with the solution of formaldehyde and acid catalyst will vary with one another, the time required increasing rapidly as the

15       temperature diminishes. When it is desired, the product may be isolated by washing and drying; preferably at a temperature of about 212°F. The products obtained according to this process are said to show no increase in wet strength and possess a high water imbibition, an increased resistance to creasing and a slight increase in affinity to some direct dyes.

20           In recent years additional methods have been devised for treating cellulosic fiber-containing products in order to impart durable crease retention, wrinkle resistance and smooth drying characteristics to these products. As discussed, formaldehyde has been crosslinked with cellulose materials to produce these products. It is also known to treat cellulose

25       materials with resins or precondensates of the urea-formaldehyde or substituted urea-formaldehyde type to produce a resin treated durable press product. As noted in U.S. patent Number 3,841,832, while formaldehyde has made a significant contribution to the cotton finishing art, the result has been far from perfect. For instance, in some cases the formaldehyde crosslinking

30       treatment has tended to lack reproducibility, since control of the

formaldehyde cross-linking reaction has been difficult. As noted in United States patent 4, 396,390, lack of reproducibility is especially true on a commercial scale.

Moreover, unacceptable loss of fabric strength has also been  
5 observed in many of the proposed aqueous formaldehyde treatment  
processes. When high curing temperatures were used with an acid or  
potential acid catalyst, excess reaction and degradation of the cotton often  
happened which considerably impaired its strength. On the other hand,  
when attempts were made to achieve reproducibility at temperatures of  
10 106°F or less, much longer reaction or finishing times were usually required,  
rendering the process economically relatively unattractive. A solution to this  
is set forth in United States Patent 4,108,598, the entire disclosure of which  
is herein incorporated by reference.

15

## SUMMARY OF THE INVENTION

In accordance with the present invention it is possible to obtain good durable press properties in a cellulosic fiber-containing fabric with good strength retention with a process that produces consistent results. This invention relates to a durable press/wrinkle-free process for cellulosic fiber-containing fabrics and more particularly to a process which utilizes formaldehyde and catalysts with silicone elastomers to impart wrinkle resistance to the cellulosic fiber-containing fabrics while reducing loss in both tensile and tear strength. This process is particularly effective on 100% cotton fabric.

## DESCRIPTION OF PREFERRED EMBODIMENTS

Such cellulosic fiber-containing fabrics include cloth made of cotton or cotton blends. There is a constant consumer demand for better treatment, that is, a more wrinkle-free product and for higher amounts of cotton in the blended fabric, or preferably, a 100% cotton fabric. There is a great demand for a wrinkle-free fabric made entirely of cotton and having good tensile and tear strength. This has been achieved and 100% cotton fabrics are treated today, but only in heavier weight pants or bottom weight fabrics. Unfortunately, the more wrinkle-free the cellulosic containing fabric is made by treatment in a formaldehyde system, the greater the loss in tear and tensile strength.

That is, as the amount of chemicals used in the treating process are increased to obtain an acceptable wrinkle resistance in the treated fabric, the loss in tear and tensile strength fall to unacceptable levels. Polyester fibers are most often blended into the cotton to form a polyester cotton blend fabric to compensate for the loss in strength of the treated cotton. Polyester in

amounts of up to 65% are commonly used. Because of the presence of polyester fibers or other synthetic fibers in the blend, these blended fabrics are sufficiently strong but do not have the comfort or feel of fabrics containing a higher amount of cotton, or most desirably, 100% cotton. The process of the present invention overcomes the disadvantages of the prior art processes and permits the presence of higher percentages of cotton in the blend and even the treatment of lighter weight or shirting weight 100% cotton fabrics to a commercially acceptable wrinkle free standard while retaining adequate strength in the fabric to also make it commercially acceptable. Commercial acceptability of the treated fabric is the ultimate goal of the process.

The durable press process of the present invention for treating cotton containing fabrics and 100% cotton fabric, comprises treating a cellulosic fiber-containing fabric with aqueous formaldehyde and a catalyst capable of catalyzing the crosslinking reaction between formaldehyde and cellulose in the presence of a silicone elastomer, heat curing the treated cellulosic fiber-containing fabric, preferably having a moisture content of more than 20% by weight, under conditions at which formaldehyde reacts with the cellulose in the presence of a catalyst and without the substantial loss of formaldehyde before the reaction of formaldehyde with cellulose to improve the wrinkle resistance of the fabric while reducing the loss in both tensile and tear strength. It is preferable that the cellulose containing fabric is in the fully swollen state.

Any silicone elastomer may be used in the present invention. Silicone elastomers are known materials. Silicone elastomers have a backbone made of silicon and oxygen with organic substituents attached to silicon atoms comprising n repeating units of the general formula:

The groups R and R<sup>1</sup> may be the same or different and includes for example, lower alkyl, such as methyl, ethyl, propyl, phenyl or any of these groups substituted by hydroxy groups, fluoride atoms or amino groups; in other words, reactive groups to cellulose.

- 5           The silicones used to make the silicone elastomers in the present invention are made by conventional processes which may include the condensation of hydroxy organosilicon compounds formed by hydrolysis of organosilicon halides. The required halide can be prepared by a direct reaction between a silicon halide and a Grignard reagent. Alternate  
10       methods may be based on the reaction of a silane with unsaturated compounds such as ethylene or acetylene. After separation of the reaction products by distillation, organosilicon halides may be polymerized by carefully controlled hydrolysis to provide the silicone polymers useful in the present invention.
- 15       For example, elastomers may be made by polymerization of the purified tetramer using alkaline catalysts at 212-302 degrees F., the molecular weight being controlled by using a monofunctional silane. Curing characteristics and properties may be varied over a wide range by replacing some methyl groups by -H, -OH, fluoroalkyl, alkoxy or vinyl groups and by  
20       compounding with fillers as would be appreciated by one of ordinary skill in the art.

- Silicone elastomers used in the present invention are high weight materials, generally composed of dimethyl silicone units (monomers) linked  
25       together in a linear chain. These materials usually contain a peroxide type catalyst which causes a linking between adjacent methyl groups in the form of methylene bridges. The presence of crosslinking greatly improves the durability of the silicone elastomer on cellulose by producing larger molecules.

30



It is also possible to produce a reactive silicone elastomer, which is one where reactive groups capable of reacting with the substrate have been added to the linear dimethyl silicone polymer. These silicones are capable of reacting both with cellulose substrates as well as with most protein fibers, and are characterized by much greater durability of the silicone polymer on the substrate, even approaching the life of the substrate.

Therefore silicone elastomers which give off reaction gases or chemicals indicating chemical reaction with the substrate are much preferred over non reactive silicone elastomer, but this is not to say that non reactive silicone elastomers cannot be used in the process. Different elastomers, by different manufacturers have all shown increases in tensile as well as tear strength, as shown in Tables I and II included herein. Elastomeric silicone polymers have been found to increase strength whereas simple emulsified silicone oils (or lubricants) do not give increases in tensile strength.

The aqueous system containing formaldehyde, an acid catalyst, silicone elastomer and a wetting agent may be padded on the fabric to be treated, preferably to insure a moisture content of more than 20% by weight on the fabric, and then the fabric cured. The padding technique is conventional to the art and generally comprises running the fabric through the aqueous solution which is then passed through squeezing rollers to provide a wet pick-up of about 66%. As is conventional in the art, the concentration of the reactants in the aqueous solution are adjusted to provide the desired amount of reactants on the weight of the fabric (OWF).

It is possible to use unexpected high temperatures which allow the crosslinking reaction to take place before the loss of formaldehyde is great enough to affect the process and provide inadequate treatment. In accordance with this aspect of the invention, the padded fabric may be immediately plunged into a heating chamber at from about 300 to about 325°F. This is an important commercial aspect of the invention as it enables

continuous processing on a commercial scale at speeds of 100-200 yards per minute. It must be appreciated, that this process is designed for commercial applications which are demanding in that the process must be commercially viable.

5           This may also be accomplished by curing at a low temperature with an active catalyst. It is also possible to use any combination of techniques which prevent the substantial loss of formaldehyde during the curing. For example, a low temperature may be used in combination with an aqueous formaldehyde solution. It would also be possible to use a pressurized system  
10       wherein the pressure is greater than atmospheric, thereby preventing the substantial loss of formaldehyde before the formaldehyde crosslinks with the cellulosic fiber-containing fabric being treated.

          In addition the process of the present invention uses less formaldehyde than other known processes. Shirting fabrics treated in  
15       accordance with the process of the present invention contain approximately 1000 ppm after treatment before steaming on a shirting fabric as compared to 3000 ppm+ by another crosslinking process on a similar shirting fabric. Tests have shown that continuously running steaming chambers to which the treated fabric is exposed should effectively remove residual formaldehyde to  
20       concentrations as low as 200 ppm. This is also an important aspect of the present invention in view of consumers concern about the presence of formaldehyde in their purchased garments. It is also possible to wash fabrics either continuously or in batch washers. Both approaches remove essentially all of the formaldehyde.

25           It is known to add to the fabric a polymeric resinous additive that is capable of forming soft film. For example, such additives may be a latex or fine aqueous dispersion of polyethylene, various alkyl acrylate polymers, acrylonitrile-butadiene copolymers, deacetylated ethylene-vinyl acetate copolymers, polyurethanes and the like. Such additives are well known to  
30       the art and are generally commercially available in concentrated aqueous

latex form. Such a latex is diluted to provide about 1 to 3% polymer solids in the aqueous catalyst-containing padding bath before the fabric is treated therewith. One known softener which was virtually the softener of choice in the durable press process using resin treatment or formaldehyde crosslinking was high density polyethylene, Mykon HD. It has been unexpectedly  
5 discovered that the substitution of a silicone elastomer for high density polyethylene significantly reduces the loss in tear strength of the treated fabric after washing as well as providing better control of the process as may be seen from the examples. The importance of good control of the process  
10 is essential to a commercially viable process to provide a consistent product from run to run which is not adversely affected by variations in atmospheric pressure, humidity and the like.

As the cellulosic fiber-containing fabric which may be treated by the present process there can be employed various natural cellulosic fibers and  
15 mixtures thereof, such as cotton and jute. Other fibers which may be used in blends with one or more of the above-mentioned cellulosic fibers are, for example, polyamides (e.g., nylons), polyesters, acrylics (e.g., polyacrylonitrile), polyolefins, polyvinyl chloride, and polyvinylidene chloride. Such blends preferably include at least 35 to 40% by weight, and most  
20 preferably at least 50 to 60% by weight, of cotton or natural cellulose fibers.

The fabric may be a resinated material but preferably it is unresinated; it may be knit, woven, non-woven, or otherwise constructed. After processing, the formed wrinkle resistant fabric will maintain the desired configuration substantially for the life of the fabric. In addition, the fabric will  
25 have an excellent wash appearance even after repeated washings.

This invention is not dependent upon the limited amounts of moisture to control the crosslinking reaction since the crosslinking reaction is most efficient in the most highly swollen state of the cellulose fiber. Lesser amounts of moisture may be used but are less preferred.

However, the silicone elastomer must be present in a sufficient amount to reduce the loss of tensile and tear strength in the fabric normally associated with the treatment of the same fabric in a prior art treatment process which may include the use of softeners such as Mykon HD. The formulation and process of the present invention may be adjusted to meet specific commercial requirements for the treated fabric. For example, formaldehyde and the catalyst concentration may be increased to provide better treatment; then the concentration of the softener is also increased to combat the loss of tear strength caused by the increased amount of catalyst used in the process. This lends itself to computerized control of the systems for treating various fabrics and allows variation in the treatment of different fabrics, which is another advantage of the process of the present invention.

While silicone oils are known as silicone softeners and have found some use in fabric treatment, they suffer serious disadvantages in having a strong tendency to produce non-removable spots. However, the particular silicone elastomer used in the process of the present invention completely overcomes these problems.

Blended fabrics to be treated in accordance with the present invention are immersed in a solution to provide a pick up or on the weight of fabric (OWF) of about 3 % formaldehyde, 1 % of catalyst, 1% of the silicone elastomer. This requires a pickup of about 66% by weight of the aqueous formulation to achieve the above stated percentage of reactants on the fabric. However, when treating 100% cotton fabric chemical concentrations must be increased so that 5% formaldehyde OWF, about 2% catalyst and about 2% elastomer padded onto the fabric. This is contrary to the prior art attempts to treat 100% cotton where the concentration of reactants were decreased because of the loss of strength due to the treatment process. The curing temperature may be about 300° F. In fact, the padded fabric may be plunged into a oven or heating chamber at 300°F.

The formaldehyde concentration may be varied as would be appreciated by one of ordinary skill in the art. The process includes the use of formaldehyde in the form of an aqueous solution having a concentration of 0.5% to 10%, by weight.

- 5 The preferred formaldehyde concentration on the fabric is from 1.5% to 7% based on the weight of the fabric.

The catalyst used in the process includes fluorosilicic acid for mild reactions and is applicable to blend fabrics. On heavyweight, all-cotton fabrics, or shirting fabrics, a catalyst such as magnesium chloride spiked with  
10 citric acid can be used, which is a commercially available catalyst Freecat No. 9, as is a similar catalyst which contains aluminum/magnesium chloride.

During the crosslinking reaction at the curing stage, moisture is given up from the fabric as the crosslinking occurs, resulting in a decrease in the moisture content of the fabric. In fabrics having a moisture content of 20%  
15 or less, this tends to lower the effectiveness of the crosslinking reaction requiring higher concentrations of formaldehyde. In a preferred aspect of the present invention, moisture is given up from a high level, that is, greater than 20%, preferably greater than 30%, e.g., from 60-100% or more, and the crosslinking is optimized. Moisture, which is so difficult to control, is not a  
20 problem in the present invention. Of course, water is not allowed to be present in so much of an excess as to cause the catalyst to migrate on the fabric.

All results reported in the following examples were obtained by the following standard methods:

- 25 1. Appearance of Fabrics after Repeated Home Launderings: AATCC Test Method 124-1992
2. Tensile Strength: ASTM :Test Method D-1682-64 (Test 1C)
3. Tear Strength: ASTM : Test Method D-1424-83 Falling Pendulum Method
- 30 4. Shrinkage: AATCC Test Method 150-1995

5. Wrinkle Recovery of Fabrics: Recovery Angle Method:

AATCC Test Method 66-1990 which provides the DP value.

In determining the DP value for the fabrics, a visual comparative test is performed under controlled lighting conditions in which the amount of wrinkles in the treated fabric is compared with the amount of wrinkles present on pre-wrinkled plastic replicas. The plastic replicas have various degrees of wrinkles and range from a value of 1 DP for a very wrinkled fabric to 5.0 DP for a flat wrinkle free fabric. The higher the DP value, the better. For a commercially acceptable wrinkle free fabric, a DP value of 3.5 is desired but rarely achieved. As would be appreciated by one of ordinary skill in the art, the difference between a DP of 3.50 and 3.25 is significant. At DP 3.50 all wrinkles are rounded and disappearing. At DP 3.25 all wrinkles are still visible and show sharp creases. The goal for commercial acceptance is a DP of 3.50 with a filling tensile strength 25 pounds and a filling tear strength of 24 ounces. Of equal or even greater importance to these properties is that the process must be consistently reproducible on an industrial scale.

In all of the following examples a non-ionic wetting agent was used as is conventional to the art. The wetting agent was used in an amount of about 0.1% by weight. The wetting agent used in all of the examples was an alkyl aryl polyether alcohol such as Triton X-100. The wetting agent is used to cause complete wetting by the aqueous treating solution of the fibers in the fabric.

All of the samples were run on all-cotton fabrics which are the most difficult to treat because of the severe loss in tensile and tear strength, which causes the treated fabric to be commercially unacceptable. The normal industry standard for tear and tensile strength for an all cotton shirting fabric is characterized by having a filling tensile strength of 25 pounds and a filling tear strength of 24 ounces. The cotton fabric must meet and/or exceed this standard. The test conditions are set forth in the table.

The silicone elastomer was the commercially available softener Sedgefield Elastomer Softener ELS, which is added as an opaque white liquid which contains from 24-26% silicone, has a pH of from 5.0-7.0 and is readily dilutable with water. When used in the present invention, this product  
5 produced DP values at catalyst concentrations of 0.8%, whereas with the Mykon HD, a catalyst concentration of 2.0% was required to give a DP value of 3.50 after 1 washing and 3.25 after 5 washings.

The tensile strength with a catalyst concentration of 0.8% and tear strength are significantly and unexpectedly higher than the 2.0% catalyst  
10 required with Mykon HD to give equal DP results. Catalyst concentration of 1.0% ELS is recommended to ensure a margin of safety, such that any variation in treatment will be well within accepted specifications.

The following examples are being presented not as limitations but to illustrate and provide a better understanding of the invention. In order to  
15 confirm the fact that formaldehyde was being lost from the conventional processes, experiments were conducted in which the fabric was heated very quickly by very hot air as in the conventional processes as well as in accordance with the present invention.

#### 20 Example 1

As indicated, it is possible to cure with a high enough temperature that the crosslinking reaction is achieved before sufficient formaldehyde is lost preventing good treatment. In this experiment, 100% cotton oxford shirting was padded with formaldehyde (37%) at a concentration of 5.0% OWF, 0.8  
25 % OWF of Freecat #9 Accelerator manufactured by Freedom Textile Chemicals Co. and 1.5 % OWF of a silicone elastomeric softener, Sedgesoft ELS manufactured by Sedgefield Specialties, to a pickup of approximately 60-70%. The sample was then dried and cured while under tension in an air circulating oven set at 300°F. for 10 minutes.

30

## Example 2

Another sample of the same fabric as used in Example 1 was padded with a similar solution differing only in that the catalyst Accelerator #9 was 1.0% OWF. Otherwise the sample was treated precisely the same.

5

## Example 3

Another sample of the same fabric as used in Example 1 was padded with a similar solution differing only in that the catalyst Accelerator #9 was 2.0% OWF. Otherwise the sample was treated precisely the same.

10

## Example 4

Another sample of the same fabric as used in Example 1 was padded with a similar solution differing only in that the catalyst Accelerator #9 was 0.4% OWF, and Mykon HD was substituted for the Sedgesoft ELS elastomeric Softener. Otherwise the sample was treated precisely the same.

15

## Example 5

Another sample of the same fabric as used in Example 1 was padded with a similar solution differing only in that the catalyst Accelerator #9 was 0.8% OWF, and Mykon HD was substituted for the Sedgesoft ELS elastomeric Softener. Otherwise the sample was treated precisely the same.

20

## Example 6

Another sample of the same fabric as used in Example 1 was padded with a similar solution differing only in that the catalyst Accelerator #9 was 1.0% OWF, and Mykon HD was substituted for the Sedgesoft ELS elastomeric Softener. Otherwise the sample was treated precisely the same.

25

30



## Example 7

Another sample of the same fabric as used in Example 1 was padded with a similar solution differing only in that the catalyst Accelerator #9 was 1.5% OWF, and Mykon HD was substituted for the Sedgesoft ELS elastomeric Softener. Otherwise the sample was treated precisely the same.

## Example 8

Another sample of the same fabric as used in Example 1 was padded with a similar solution differing only in that the catalyst Accelerator #9 was 2.0% OWF, and Mykon HD was substituted for the Sedgesoft ELS elastomeric Softener. Otherwise the sample was treated precisely the same.

## Example 9

A sample of the same fabric was washed in a home washer and tumble dried, but not treated with any crosslinking process.

## Example 10

Another sample of the same fabric served as an untreated, unwashed control.

TABLE NO. I

Sedgefield Silicone Elastomeric Softener ELS vs. MykonHD, High Density Polyethylene

Sample Treatment Date: May 1996

Fabric: New Cherokee 100% Cotton Oxford Shirting

Example No	Fabric Type	CH <sub>2</sub> O %OWF	Cat # 9 %OWF	Softener	Amount %OWF	Cure Temp °F.	Cure Time Min	Tensile' W X F	Tear' W X F	Shrink 1 Wash W X F %	DP 1 Wash	Shrink 5 Washes W X F %	DP 5 Washes
1	Oxford	5.0	0.8	ELS	1.5	300	10	45.3 X 45.0	59.4 X 45.2	1.08 X 0.58	3.50	1.50 X 0.83	3.50
2	Oxford	5.0	1.0	ELS	1.5	300	10	43.7 X 41.3	48.5 X 42.9	0.75 X 0.58	3.50	1.25 X 0.67	3.50
3	Oxford	5.0	2.0	ELS	1.5	300	10	30.0 X 29.0	28.9 X 25.5	0.75 X 0.67	3.50	0.92 X 0.75	3.50
4	Oxford	5.0	0.4	Mykon HD	1.5	300	10	61.8 X 69.8	103.8 X 79.5	2.00 X 1.42	2.0	2.50 X 1.08	2.00
5	Oxford	5.0	0.8	Mykon HD	1.5	300	10	53.0 X 58.2	72.9 X 53.4	1.67 X 1.08	2.75	1.83 X 0.92	2.50
6	Oxford	5.0	1.0	Mykon HD	1.5	300	10	47.2 X 47.2	60.3 X 42.4	1.17 X 0.83	3.25	1.17 X 0.67	2.50
7	Oxford	5.0	1.5	Mykon HD	1.5	300	10	39.3 X 37.5	36.6 X 28.6	0.83 X 0.67	3.25	0.75 X 0.33	3.00
8	Oxford	5.0	2.0	Mykon HD	1.5	300	10	34.7 X 35.0	27.8 X 25.5	0.75 X 0.67	3.50	0.75 X 0.42	3.25
9	Control Unwashed	—	—	—	—	—	—	74.3 X 99.0	120.1 X 133.2	2.00 X 1.58	< 1.0	4.42 X 1.83	< 1.0
10	Control Washed	—	—	—	—	—	—	71.7 X 100.8	35.7 X 63.9	—	—	—	—

1. Evaluated after treatment but before washing.

It is clear in Table No. I that samples treated with the elastomeric softener produced higher degrees of durable press than any of the samples treated with Mykon HD. Tensile Strengths are similar as is shrinkage for each degree of treatment.

5           In another experiment, the results shown in Table No. II, samples of 100% cotton oxford shirting were padded with two concentrations of formaldehyde 3.0 and 5.0% OWF, each concentration also treated with three concentrations of Accelerator #9 Catalyst, 0.8, 1.0, and 2.0% . In one half of the samples, Sedgesoft  
10       ELS was applied and in the other half Mykon HD was used as the softener. Both softeners were applied at 1.5% OWF. Each of the samples were padded with the respective solutions shown in Table No. II, then cured at 300°F. for 10 minutes under tension. All samples were treated in precisely the same way, intervals were  
15       timed.

          It is clearly seen in Table II (Example 11 to Example 22 and the control) that after 5 washes, the Sedgesoft ELS samples have almost twice the tear strength of the Mykon HD samples without exception. In addition, again seen, the DP values are higher  
20       indicating better smoothness.

**TABLE NO. II**  
**Comparison of Softeners, Sedgesoft ELS vs. Mykon HD**  
**Silicone Polymer Emulsion**  
**Polyethylene Emulsion**  
**Tensile, Filling: 25 lbs.; Tear, Filling: 24 oz.**

Treatment:  
 Sedgesoft ELS:  
 Mykon HD:

Specification Strength:

Example No.	Fabric New Charolles Oxford Shirting	CH <sub>2</sub> O %OWF	Cal #9 %OWF	Softener Type	Softener Amt. %OWF	Cure/Time F./Min.	Tensile' Lbs. W X F	Tear' Oz. W X F	Shrink 1 Wash W X F %	DP 1 Wash	Shrink 5 Wash W X F %	DP 5 Washes	Tensile' 5 Washes W X F	Tear' 5 Washes W X F
11	100% Cotton	3.0	0.8	ELS	1.5	300/10	51.9 X 53.3	66.2 X 49.0	2.50 X 1.42	2.75	3.50 X 1.75	2.75	52.2 X 60.0	54.2 X 66.8
12	100% Cotton	3.0	1.0	ELS	1.5	300/10	43.7 X 39.7	44.0 X 36.6	1.83 X 1.42	3.00	2.500 X 1.67	2.90	47.0 X 53.2	42.9 X 40.6
13	100% Cotton	3.0	2.0	ELS	1.5	300/10	31.8 X 29.3	27.5 X 21.0	1.25 X 1.17	3.25	1.75 X 1.42	3.00	34.2 X 34.5	26.6 X 24.1
14	100% Cotton	3.0	0.8	HD	1.5	300/10	54.8 X 55.7	75.2 X 50.8	2.00 X 1.58	2.75	2.92 X 2.00	2.00	56.8 X 65.8	29.4 X 32.3
15	100% Cotton	3.0	1.0	HD	1.5	300/10	49.7 X 46.7	60.9 X 41.1	1.75 X 1.17	3.00	2.50 X 1.75	2.50	54.0 X 60.0	27.8 X 29.8
16	100% Cotton	3.0	2.0	HD	1.5	300/10	38.2 X 34.2	29.4 X 23.3	1.17 X 1.255	3.25	1.67 X 1.33	3.00	35.5 X 39.6	19.6 X 19.9
17	100% Cotton	5.0	0.8	ELS	1.5	300/10	46.7 X 44.0	56.4 X 35.4	1.92 X 1.25	2.75	2.42 X 1.33	2.90	47.0 X 59.5	50.3 X 65.5
18	100% Cotton	5.0	1.0	ELS	1.5	300/10	43.2 X 38.2	40.6 X 30.5	1.58 X 1.08	3.00	1.91 X 1.00	3.00	36.0 X 51.6	43.3 X 58.0
19	100% Cotton	5.0	2.0	ELS	1.5	300/10	30.8 X 27.3	26.8 X 27.5	1.08 X 0.92	3.25	0.75 X 0.75	3.25	30.0 X 37.3	28.7 X 33.2
20	100% Cotton	5.0	0.8	HD	1.5	300/10	51.5 X 49.0	63.2 X 43.6	2.00 X 1.87	2.50	2.58 X 1.67	2.75	49.7 X 67.5	28.7 X 42.9
Example No.	Fabric New Charolles Oxford Shirting	CH <sub>2</sub> O %OWF	Cal #9 %OWF	Softener Type	Softener Amt. %OWF	Cure/Time F./Min.	Tensile' Lbs. W X F	Tear' Oz. W X F	Shrink 1 Wash W X F %	DP 1 Wash	Shrink 5 Wash W X F %	DP 5 Washes	Tensile' 5 Washes W X F	Tear' 5 Washes W X F
21	100% Cotton	5.0	1.0	HD	1.5	300/10	44.0 X 48.0	40.0 X 31.8	1.67 X 1.58	2.50	2.00 X 1.33	3.00	49.3 X 52.0	29.8 X 37.7

**TABLE NO. II (Continued)**

22	100% Cotton	5.0	2.0	HD	1.5	300/10	33.2 X 32.5	28.6 X 21.0	1.08 X 0.92	3.00	1.08 X 1.00	3.15	26.3 X 41.0	17.6 X 19.9
Washed Control (5 Washes)														
100% Cotton							74.1 X 106.7	77.4 X 103.8	2.92 X 1.67	<1.0	3.30 X 1.00	<1.0	70.1 X 109.7	37.7 X 59.4

1. Evaluated after treatment but before washing.
2. Evaluated after 5 washings.

What is claimed is:

1. A durable press process for cellulosic fiber-  
containing fabrics comprising treating a cellulose fiber-containing  
5 fabric with formaldehyde, a catalyst capable of catalyzing the  
crosslinking reaction between formaldehyde and cellulose, and  
an effective amount of silicone elastomer, heat curing said  
treated cellulosic fiber-containing fabric under conditions at  
which formaldehyde reacts with cellulose in the presence of the  
10 catalyst and silicone elastomer, without a substantial loss of  
formaldehyde before the reaction of the formaldehyde with the  
cellulose to improve the wrinkle resistance of the fabric while  
reducing loss in tear and tensile strength.
- 15 2. The process of claim 1 wherein the heat curing is  
at a temperature which prevents the substantial loss of  
formaldehyde during curing.
- 20 3. The process of claim 1 wherein the heat curing  
step is carried out at a high enough temperature to allow the  
crosslinking step to occur before sufficient formaldehyde leaves  
the fabric and affects the process.
- 25 4. The process of claim 1 wherein the fabric being  
cured has a moisture content of more than 20% by weight.
5. The process of claim 2 wherein the heat curing is  
over a temperature of from 100°F to 350°F.

6. The process in claim 2 where the heat curing is carried out in the preferred range of 250 to 325°F.

5 7. The process of claim 1 wherein said fabric is heat cured by gradually increasing the temperature.

8. The process of claim 5 wherein the heat curing is over a temperature of from 100°F to 300°F.

10 9. The process in claim 1 where the formaldehyde is in the form of an aqueous solution of formaldehyde having a concentration of 0.5% to 10%.

15 10. The process in claim 1 where the preferred formaldehyde concentration range is from 1.5% to 7% on the weight of the fabric.

11. The process of claim 1, wherein the fabric is 100% cotton shirting.

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## INTERNATIONAL SEARCH REPORT

International application No.  
PCT/US98/09367

## A. CLASSIFICATION OF SUBJECT MATTER

IPC(6) : D06M 13/127

US CL : 8/116.4, 115.7; 427/389.9, 394

According to International Patent Classification (IPC) or to both national classification and IPC

## B. FIELDS SEARCHED

Minimum documentation searched (classification system followed by classification symbols)

U.S. : 8/116.4, 115.7; 427/389.9, 394

Documentation searched other than minimum documentation to the extent that such documents are included in the fields searched

Electronic data base consulted during the international search (name of data base and, where practicable, search terms used)

## C. DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
X ----- Y	US 3,663,974 A (WANTANABE et al) 23 May 1972, col. 2, lines 8-44; and examples 1-3 and 12.	1-3, 5, 8-11 ----- 4, 6, 7
A	US 2,243,765 A (MORTON) 27 May 1941, col. 1, lines 1-32.	1-11
A	US 3,812,201 A (BEY) 21 May 1974, col. 1, lines 10-14.	1-11
A	US 4,108,598 A (PAYET) 22 August 1978, col. 2, lines 23-33.	1-11
A	US 4,269,603 A (WORTH) 26 May 1981, col. 2, lines 21-32.	1-11



Further documents are listed in the continuation of Box C.



See patent family annex.

*.	Special categories of cited documents:	*T*	later document published after the international filing date or priority date and not in conflict with the application but cited to understand the principle or theory underlying the invention
*A*	document defining the general state of the art which is not considered to be of particular relevance	*X*	document of particular relevance; the claimed invention cannot be considered novel or cannot be considered to involve an inventive step when the document is taken alone
*E*	earlier document published on or after the international filing date	*Y*	document of particular relevance; the claimed invention cannot be considered to involve an inventive step when the document is combined with one or more other such documents, such combination being obvious to a person skilled in the art
*L*	document which may throw doubts on priority claim(s) or which is cited to establish the publication date of another citation or other special reason (as specified)	*A*	document member of the same patent family
*O*	document referring to an oral disclosure, use, exhibition or other means		
*P*	document published prior to the international filing date but later than the priority date claimed		

Date of the actual completion of the international search

19 JUNE 1998

Date of mailing of the international search report

10 AUG 1998

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## INTERNATIONAL SEARCH REPORT

International application No.  
PCT/US98/09367

## C (Continuation). DOCUMENTS CONSIDERED TO BE RELEVANT

Category*	Citation of document, with indication, where appropriate, of the relevant passages	Relevant to claim No.
A	US 4,369,390 A (HENDRIX et al) 02 August 1983, col. 2, lines 1-10.	1-11

Form PCT/ISA/210 (continuation of second sheet)(July 1992)\*

